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ANTISTATIC COMPOSITION

Field of the Invention

This invention relates to compositions comprising at least one polymer and at least one antistatic agent. This invention further relates to
10 fibers, films, fabrics, coatings, and molded or blown articles comprising the compositions. In other aspects, this invention also relates to novel compounds that are useful as antistatic agents and to processes for imparting antistatic characteristics to substrates.

15
Background of the Invention

Electrostatic charge buildup is responsible for a variety of problems in the processing and use of many industrial products and materials. Electrostatic
20 charging can cause materials to stick together or to repel one another. This is a particular problem in fiber and textile processing. In addition, static charge buildup can cause objects to attract dirt and dust, which can lead to fabrication or soiling problems
25 and can impair product performance.

Sudden electrostatic discharges from insulating objects can also be a serious problem. With photographic film, such discharges can cause fogging and the appearance of artifacts. When flammable
30 materials are present, a static electric discharge can serve as an ignition source, resulting in fires and/or explosions.

Static is a particular problem in the electronics industry, since modern electronic devices
35 are extremely susceptible to permanent damage by static electric discharges. The buildup of static charge on

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insulating objects is especially common and problematic under conditions of low humidity and when liquids or solids move in contact with one another (tribocharging).

5 Static charge buildup can be controlled by increasing the electrical conductivity of a material. This can be accomplished by increasing ionic or electronic conductivity. The most common means of controlling static accumulation today is by increasing electrical conductivity through moisture to the material. This is commonly achieved by adding moisture to the surrounding air (humidification) or by use of hygroscopic antistatic agents, which are generally referred to as humectants since they rely on the adsorption of atmospheric moisture for their effectiveness. Most antistatic agents operate by dissipating static charge as it builds up; thus, static decay rate and surface conductivity are common measures of the effectiveness of antistatic agents.

10 Antistatic agents can be applied to the surface (external antistat) or incorporated into the bulk (internal antistat) of an otherwise insulating material. Internal antistats are commonly employed in polymers such as plastics. Generally, internal antistats are mixed directly into a molten polymer during melt processing. (Typical polymer melt processing techniques include molding, melt blowing, melt spinning, and melt extrusion.) Relatively few antistatic agents have the requisite thermal stability to withstand polymer melt processing temperatures, which can be as high as 250 to 400°C or more. Since static buildup is typically a surface phenomenon, internal antistats that are capable of migrating to and enriching the surface of a material are generally most effective.

Known antistatic agents cover a broad range of chemical classes, including organic amines and amides, esters of fatty acids, organic acids, polyoxyethylene derivatives, polyhydric alcohols, 5 metals, carbon black, semiconductors, and various organic and inorganic salts. Many are also surfactants and can be neutral or ionic in nature.

Many low molecular weight, neutral antistats have sufficiently high vapor pressures that they are 10 unsuitable for use at high temperatures, as in polymer melt processing, due to material losses that occur via evaporation. Many other neutral antistats have insufficient thermal stability to survive polymer melt processing or other high temperature processing 15 conditions.

Most nonmetallic antistats are humectants that rely on the adsorption and conductivity of water for charge dissipation. Thus, their effectiveness is typically diminished at low atmospheric humidity. 20 Since many of these antistatic agents are also water soluble, they are easily removed by exposure of the material to water (as in washing) and are therefore not very durable. Water associated with hygroscopic antistatic agents can be a particular problem during 25 polymer melt processing, since the water tends to vaporize rapidly at melt processing temperatures. This leads to the undesirable formation of bubbles in the polymer and can cause screw slippage in extrusion equipment.

30 Quaternary ammonium salts are well known in the art to be useful antistatic agents. They can be solid or liquid, the most common being halide or methanesulfonate salts. The salts provide excellent antistatic performance but suffer from limited thermal 35 stability and are generally hygroscopic. Thus, they

are not capable of withstanding the high temperature processing conditions required for many high performance thermoplastic resins.

Metal salts of inorganic, organic, and fluoroorganic anions have also shown proven utility as antistatic agents in certain polymer compositions. Alkali metal salts are most commonly employed, due to cost and toxicity considerations and to the high affinity of alkali metal cations, especially lithium, for water. However, most metal salts provide insufficient thermal stability under high temperature processing conditions and are not compatible with polymers of moderate to low polarity, such as polypropylene, polyester, and polycarbonate. This incompatibility can result in inadequate antistat performance and/or an unacceptable reduction in physical properties or transparency in a finished polymeric article. Consequently, the utility of metal salts as internal antistatic agents is generally limited to highly polar and/or hydrophilic polymer matrices cast from aqueous or organic solution at relatively low temperatures.

Furthermore, since many metal salts are corrosive towards metals and electronic components, they are unsuitable for applications where they may come into contact with such surfaces. Known hydrophilic metal salts and quaternary ammonium salts generally suffer all the disadvantages of other humectant antistatic agents (vide supra).

Thus, there remains a need in the art for antistatic agents that exhibit a superior balance of high thermal stability, hydrophobicity, low volatility, low corrosivity toward metals and electronic components, durability, and polymer compatibility, and that can impart good antistatic performance to a

variety of insulating materials over a wide range of humidity levels.

Summary of the Invention

Briefly, in one aspect, this invention provides an antistatic composition comprising or consisting essentially of a melt blend of (a) at least one ionic salt consisting of a nonpolymeric nitrogen onium cation (for example, a quaternary ammonium ion) and a weakly coordinating fluoroorganic anion, the conjugate acid of the anion being a superacid (for example, a bis(perfluoroalkanesulfonyl)imide ion); and (b) at least one thermoplastic polymer. As used herein, the term "melt blend" means a blend that has been prepared by melt processing technique(s), and the term "onium" means a positively charged ion having at least part of its charge localized on at least one nitrogen atom. Preferably, the Hammett acidity function, H_0 , of the conjugate acid of the anion is less than about -10.

It has been discovered that the above-described ionic salts can be used as additives (internal antistats) or topical treatments (external antistats) to impart antistatic characteristics to polymers or other insulating materials. These ionic salts are surprisingly effective at dissipating the static charge that can accumulate in an otherwise insulating substrate such as a polymer film or fabric. For example, when incorporated as polymer melt additive in polypropylene melt-blown nonwoven fabric, certain preferred salts impart static dissipation rates that are as good or better than those of any known antistatic agents under the same static decay test conditions. The ionic salts used in the composition of the invention are effective, even without the presence

of a conductivity enhancing additive (for example, a lithium salt or a polar organic solvent), and thus compositions consisting essentially of salt and insulating material surprisingly exhibit good antistatic characteristics.

In addition, the ionic salts used in the composition of the invention exhibit surprisingly high thermal stabilities. The salts (surprisingly, even the quaternary ammonium salts) remain stable at temperatures up to 300-500°C (often, and preferably, at temperatures greater than 350°C) and thus are particularly well-suited for use as polymer melt additives (incorporated in host polymer through high temperature melt processing) and in applications where the use temperatures are very high. The salts are also nonvolatile (having essentially no vapor pressure), nonflammable, and can be utilized under normal processing and use conditions without the emission of potentially harmful vapors and without the gradual declines in antistat performance that result from evaporative loss.

The ionic salts used in the composition of the invention are compatible with a variety of polymers. Many of the salts are also hydrophobic (immiscible with water), and thus their antistatic performance is relatively independent of atmospheric humidity levels and durable even under exposure to aqueous environments. Preferred ionic salts are liquid at room temperature (for example, at about 25°C) and above.

The ionic salts used in the composition of the invention therefore meet the need in the art for antistatic agents that exhibit a superior balance of high thermal stability, hydrophobicity, low volatility, durability, and polymer compatibility, while imparting

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good antistatic performance to a variety of insulating materials over a wide range of humidity levels.

In other aspects, this invention also provides fiber, fabric, film, a coating, and a molded or blown article comprising the composition of the invention; novel compounds useful as antistatic agents; and processes for imparting antistatic characteristics to a substrate, for example, by bulk addition or by topical treatment.

10

Detailed Description of the Invention

Ionic salts suitable for use in the antistatic composition of the invention are those that consist of a nonpolymeric nitrogen onium cation and a weakly coordinating fluoroorganic (either fully fluorinated, that is perfluorinated, or partially fluorinated) anion. The nitrogen onium cation can be cyclic (that is, where the nitrogen atom(s) of the cation are ring atoms) or acyclic (that is, where the nitrogen atom(s) of the cation are not ring atoms but can have cyclic substituents). The cyclic cations can be aromatic, unsaturated but nonaromatic, or saturated, and the acyclic cations can be saturated or unsaturated.

The cyclic cations can contain one or more ring heteroatoms other than nitrogen (for example, oxygen or sulfur), and the ring atoms can bear substituents (for example, hydrogen, halogen, or organic groups such as alkyl, alicyclic, aryl, alkalicyclic, alkaryl, alicyclicalkyl, aralkyl, aralicyclic, and alicyclicaryl groups). Separate alkyl substituents can be joined together to constitute a unitary alkylene radical of from 2 to 4 carbon atoms forming a ring structure converging on nitrogen.

Organic substituents can contain one or more

heteroatoms such as, for example, nitrogen, oxygen, sulfur, phosphorus, or halogen (and thus can be fluoroorganic in nature).

5 The acyclic cations can have at least one (preferably, at least two; more preferably, at least three; most preferably, four) nitrogen-bonded organic substituents or R groups, with the remaining substituents being hydrogen. The R groups can be cyclic or acyclic, saturated or unsaturated, aromatic
10 or nonaromatic, and can contain one or more heteroatoms such as, for example, nitrogen, oxygen, sulfur, phosphorus, or halogen (and thus can be fluoroorganic in nature).

15 Preferably, the nitrogen onium cation is acyclic, saturated cyclic, or aromatic. More preferably, the cation is acyclic or aromatic. Most preferably, the cation is aromatic for stability reasons.

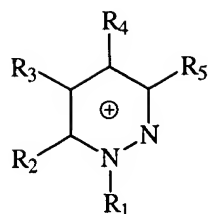
20 Preferred acyclic nitrogen onium cations are quaternary or tertiary (most preferably, quaternary) ammonium ions. The quaternary and tertiary ammonium ions are preferably of low symmetry (having at least two, preferably at least three, different nitrogen-bonded organic substituents or R groups as defined
25 above) and more preferably contain at least one hydroxyl group in at least one nitrogen-bonded organic substituent. Most preferred acyclic nitrogen onium cations are those described below for the ionic salts of Formula I.

30 Preferred aromatic nitrogen onium cations are those selected from the group consisting of

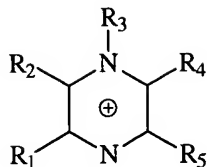
$$\begin{array}{c}
 R_1 \\
 | \\
 R_6 \quad \diagdown \quad \text{C} \quad \diagup \quad R_2 \\
 | \quad \quad \quad | \\
 R_5 \quad \diagdown \quad \text{C} \quad \diagup \quad R_3 \\
 | \quad \quad \quad | \\
 N^+ \\
 | \\
 R_4
 \end{array}$$
$$\begin{array}{c}
 R_4 \\
 | \\
 R_3 - C - C - R_5 \\
 | \quad | \\
 R_2 - N^+ - N \\
 | \\
 R_1
 \end{array}$$
*c1nc(R2)c(R3)n(R4)c1R5
$$\begin{array}{c}
 \text{R}_3 \\
 | \\
 \text{R}_2 - \text{C} - \text{N} - \text{C} - \text{R}_4 \\
 | \quad \quad | \\
 \text{R}_1 - \text{C} - \text{N} - \text{C} - \text{R}_5 \\
 | \quad \quad | \\
 \text{R}_1 \quad \text{R}_5
 \end{array}$$
$$\begin{array}{c} R_4 \quad R_5 \\ \diagdown \quad / \\ C \\ / \quad \backslash \\ N^+ \quad N \\ / \quad \backslash \\ R_3 \quad R_1 \\ | \\ R_2 \end{array}$$
R4C1C(R5)N(R)C(R2)C1*N1C(R4)C(R3)S1C(R2)*N1C(=O)C(=O)C(=O)C1*
$$\begin{array}{c} \text{R}_1 \\ | \\ \text{N}-\text{N} \\ | \quad | \\ \text{R}_4 \quad \oplus \quad \text{R}_2 \\ | \\ \text{N} \\ | \\ \text{R}_3 \end{array}$$

wherein R_1 , R_2 , R_3 , R_4 , R_5 , and R_6 are independently selected from the group consisting of H, F, alkyl groups of from 1 to about 4 carbon atoms, two said alkyl groups joined together to form a unitary alkylene radical of from 2 to 4 carbon atoms forming a ring structure converging on N, and phenyl groups; and wherein said alkyl groups, alkylene radicals, or phenyl groups can be substituted with one or more electron withdrawing groups (preferably selected from the group consisting of F-, Cl-, CF_3 -, SF_5 -, CF_3S -, $(CF_3)_2CHS$ -, and $(CF_3)_3CS$ -).

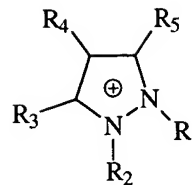
More preferred aromatic cations include those
15 selected from the group consisting of



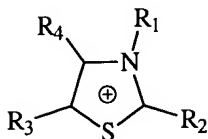
Pyridazinium



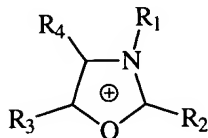
Pyrazinium



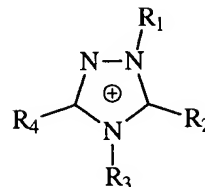
Pyrazolium



Thiazolium



Oxazolium



Triazolium

where R_1 , R_2 , R_3 , R_4 , and R_5 are as defined above.

The weakly coordinating anion is a

- 5 fluoroorganic anion, the conjugate acid of which is a superacid (that is, an acid that is more acidic than 100 percent sulfuric acid). Preferably, the Hammett acidity function, H_0 , of the conjugate acid of the anion is less than about -10 (more preferably, less than
- 10 about -12). Such weakly coordinating fluoroorganic anions include those that comprise at least one highly fluorinated alkanesulfonyl group, that is, a perfluoroalkanesulfonyl group or a partially fluorinated alkanesulfonyl group wherein all non-
- 15 fluorine carbon-bonded substituents are bonded to carbon atoms other than the carbon atom that is directly bonded to the sulfonyl group (preferably, all non-fluorine carbon-bonded substituents are bonded to carbon atoms that are more than two carbon atoms away
- 20 from the sulfonyl group).

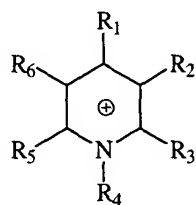
Preferably, the anion is at least about 80 percent fluorinated (that is, at least about 80 percent of the carbon-bonded substituents of the anion are fluorine atoms). More preferably, the anion is
 5 perfluorinated (that is, fully fluorinated, where all of the carbon-bonded substituents are fluorine atoms). The anions, including the preferred perfluorinated anions, can contain one or more catenary (that is, in-chain) heteroatoms such as, for example, nitrogen,
 10 oxygen, or sulfur.

Suitable weakly coordinating anions include, but are not limited to, anions selected from the group consisting of perfluoroalkanesulfonates, cyanoperfluoroalkanesulfonylamides,
 15 bis(cyano)perfluoroalkanesulfonylmethides, bis(perfluoroalkanesulfonyl)imides, bis(perfluoroalkanesulfonyl)methides, and tris(perfluoroalkanesulfonyl)methides.

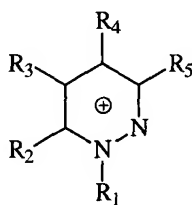
Preferred anions include
 20 perfluoroalkanesulfonates, bis(perfluoroalkanesulfonyl)imides, and tris(perfluoroalkanesulfonyl)methides. The bis(perfluoroalkanesulfonyl)imides and tris(perfluoroalkanesulfonyl)methides are more
 25 preferred anions, with the bis(perfluoroalkanesulfonyl)imides being most preferred.

The ionic salts can be solids or liquids under use conditions but preferably have melting points
 30 less than about 150°C (more preferably, less than about 50°C; most preferably, less than about 25°C). Liquid ionic salts are preferred due to their generally better static dissipative performance. The ionic salts are preferably stable at temperatures of about 325°C and
 35 above (more preferably, about 350°C and above). (In

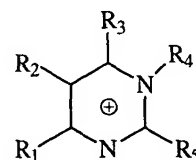
other words, the onset of decomposition of the salts is above such temperatures.) The salts are also preferably hydrophobic. Thus, a preferred class of ionic salts for use in the antistatic composition of the invention includes those that consist of (a) an aromatic nitrogen onium cation selected from the group consisting of



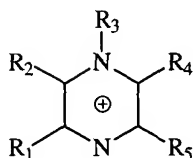
Pyridinium



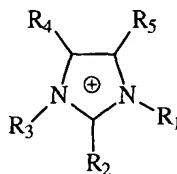
Pyridazinium



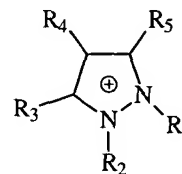
Pyrimidinium



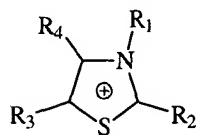
Pyrazinium



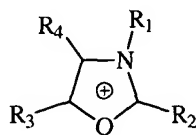
Imidazolium



Pyrazolium

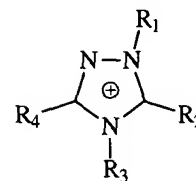


Thiazolium



Oxazolium

, and



Triazolium

- wherein R₁, R₂, R₃, R₄, R₅, and R₆ are independently selected from the group consisting of H, F, alkyl groups of from 1 to about 4 carbon atoms, two said alkyl groups joined together to form a unitary alkylene radical of from 2 to 4 carbon atoms forming a ring structure converging on N, and phenyl groups; and

wherein said alkyl groups, alkylene radicals, or phenyl groups can be substituted with one or more electron withdrawing groups (preferably selected from the group consisting of F-, Cl-, CF₃-, SF₅-, CF₃S-, (CF₃)₂CHS-, and (CF₃)₃CS-); and (b) a weakly coordinating fluoroorganic anion in accordance with the above description or a weakly coordinating anion selected from the group consisting of BF₄-, PF₆-, AsF₆-, and SbF₆-. This preferred class comprises a subclass of the hydrophobic ionic liquids described in U.S. Pat. No. 5,827,602 (Koch et al.), the description of the members of which is incorporated herein by reference.

Another preferred class of ionic salts useful in preparing the antistatic composition of the invention is the class of novel compounds represented by Formula I below



wherein each R₁ is independently selected from the group consisting of alkyl, alicyclic, aryl, alkalicyclic, alkaryl, alicyclicalkyl, aralkyl, aralicyclic, and alicyclicaryl moieties that can contain one or more heteroatoms such as, for example, nitrogen, oxygen, sulfur, phosphorus, or halogen (and thus can be fluoroorganic in nature); each R₂ is independently selected from the group consisting of hydrogen and the moieties described above for R₁; z is an integer of 1 to 4; q is an integer of 1 to 4; and X⁻ is a weakly coordinating fluoroorganic anion as described above. R₁ is preferably alkyl, and R₂ is preferably selected from the group consisting of hydrogen, alkyl, and acyl (more preferably, hydrogen or acyl; most preferably, hydrogen).

The above-described ionic salts that are useful in the antistatic composition of the invention can be prepared by ion exchange or metathesis reactions, which are well known in the art. For example, a precursor onium salt (for example, an onium halide, onium alkanesulfonate, onium alkanecarboxylate, or onium hydroxide salt) can be combined with a precursor metal salt or the corresponding acid of a weakly coordinating anion in aqueous solution. Upon combining, the desired product (the onium salt of the weakly coordinating anion) precipitates (as a liquid or solid) or can be preferentially extracted into an organic solvent (for example, methylene chloride). The product can be isolated by filtration or by liquid/liquid phase separation, can be washed with water to completely remove byproduct metal halide salt or hydrogen halide, and can then be dried thoroughly under vacuum to remove all volatiles (including water and organic solvent, if present). Similar metathesis reactions can be conducted in organic solvents (for example, acetonitrile) rather than in water, and, in this case, the salt byproduct preferentially precipitates, while the desired product salt remains dissolved in the organic solvent (from which it can be isolated using standard experimental techniques). A few of the ionic salts (for example, 1-ethyl-3-methylimidazolium trifluoromethanesulfonate, available from Sigma Aldrich, Milwaukee, Wisconsin) are commercially available.

Precursor salts or acids (for use in preparing the ionic salts) can be prepared by standard methods known in the art, and many are commercially available. Such methods include the anion precursor preparative methods described in the following references, the descriptions of which are incorporated

herein by reference: imide precursors - U.S. Patent Nos. 5,874,616 (Howells et al.), 5,723,664 (Sakaguchi et al.), 5,072,040 (Armand), and 4,387,222 (Koshar); methide precursors - U.S. Patent Nos. 5,554,664
 5 (Lamanna et al.) and 5,273,840 (Dominey); sulfonate precursors - U.S. Patent Nos. 5,176,943 (Wou), 4,582,781 (Chen et al.), 3,476,753 (Hanson), and 2,732,398 (Brice et al.); sulfonate, imide, and methide precursors having catenary oxygen or nitrogen in a
 10 fluorochemical group - U.S. Patent No. 5,514,493 (Waddell et al.); disulfone precursors - R.J. Koshar and R.A. Mitsch, J. Org. Chem., 38, 3358 (1973) and U.S. Patent No. 5,136,097 (Armand).

In general, cyano-containing methides and
 15 amides containing fluoroalkanesulfonyl groups can be prepared by the reaction of fluoroalkanesulfonyl fluorides, R_fSO_2F , with anhydrous malononitrile or cyanamide, respectively, in the presence of a non-nucleophilic base. This synthetic procedure is
 20 described in Scheme 1 of U.S. Patent No. 5,874,616 (Howells et al.) for the preparation of bis(fluoroalkanesulfonyl)imides (the description of which is incorporated herein by reference) and involves the substitution of either malononitrile or cyanamide
 25 for the fluoroalkanesulfonamide. The resulting intermediate non-nucleophilic base cation-containing methide or amide salt can be converted to the desired cation salt (typically lithium) via standard metathesis reactions well known in the art.

30 Representative examples of useful ionic salts include octyldimethyl-2-hydroxyethylammonium bis(trifluoromethylsulfonyl)imide:
 $[C_8H_{17}N^+(CH_3)_2CH_2CH_2OH \quad ^-N(SO_2CF_3)_2]$,
 octyldimethyl-2-hydroxyethylammonium
 35 perfluorobutanesulfonate:

- [C₈H₁₇N⁺(CH₃)₂CH₂CH₂OH⁻OSO₂C₄F₉],
 octyldimethyl-2-hydroxyethylammonium
 trifluoromethanesulfonate:
 [C₈H₁₇N⁺(CH₃)₂CH₂CH₂OH⁻OSO₂CF₃],
 5 octyldimethyl-2-hydroxyethylammonium
 tris(trifluoromethanesulfonyl)methide:
 [C₈H₁₇N⁺(CH₃)₂CH₂CH₂OH⁻C(SO₂CF₃)₃],
 trimethyl-2-acetoxyethylammonium
 bis(trifluoromethylsulfonyl)imide:
 10 [(CH₃)₃N⁺CH₂CH₂OC(O)CH₃⁻N(SO₂CF₃)₂],
 trimethyl-2-hydroxyethylammonium
 bis(perfluorobutanesulfonyl)imide:
 [(CH₃)₃N⁺CH₂CH₂OH⁻N(SO₂C₄F₉)₂],
 triethylammonium bis(perfluoroethanesulfonyl)imide:
 15 [Et₃N⁺H⁻N(SO₂C₂F₅)₂],
 tetraethylammonium trifluoromethanesulfonate:
 [CF₃SO₃⁻*NEt₄],
 tetraethylammonium bis(trifluoromethanesulfonyl)imide:
 [(CF₃SO₂)₂N⁻*NEt₄],
 20 tetramethylammonium
 tris(trifluoromethanesulfonyl)methide:
 [(CH₃)₄N⁺⁻C(SO₂CF₃)₃],
 tetrabutylammonium bis(trifluoromethanesulfonyl)imide:
 [(C₄H₉)₄N⁺⁻N(SO₂CF₃)₂],
 25 trimethyl-3-perfluorooctylsulfonamidopropylammonium
 bis(trifluoromethanesulfonyl)imide:
 [C₈F₁₇SO₂NH(CH₂)₃N⁺(CH₃)₃⁻N(SO₂CF₃)₂],
 1-hexadecylpyridinium
 bis(perfluoroethanesulfonyl)imide:
 30 [n-C₁₆H₃₃-cyc-N⁺C₅H₅⁻N(SO₂C₂F₅)₂],
 1-hexadecylpyridinium perfluorobutanesulfonate:
 [n-C₁₆H₃₃-cyc-N⁺C₅H₅⁻OSO₂C₄F₉],
 1-hexadecylpyridinium perfluorooctanesulfonate:
 [n-C₁₆H₃₃-cyc-N⁺C₅H₅⁻OSO₂C₈F₁₇],
 35 n-butylpyridinium bis(trifluoromethanesulfonyl)imide:

- [n-C₄H₉-cyc-N⁺C₅H₅ ⁻N(SO₂CF₃)₂],
n-butylpyridinium perfluorobutanesulfonate:
[n-C₄H₉-cyc-N⁺C₅H₅ ⁻OSO₂C₄F₉],
1,3-ethylmethylimidazolium
- 5 bis(trifluoromethanesulfonyl)imide:
[CH₃-cyc-(N⁺C₂H₂NCH)CH₂CH₃ ⁻N(SO₂CF₃)₂],
1,3-ethylmethylimidazolium nonafluorobutanesulfonate:
[CH₃-cyc-(N⁺C₂H₂NCH)CH₂CH₃ ⁻OSO₂C₄F₉],
1,3-ethylmethylimidazolium trifluoromethanesulfonate:
- 10 [CH₃-cyc-(N⁺C₂H₂NCH)CH₂CH₃ ⁻OSO₂CF₃],
1,3-ethylmethylimidazolium hexafluorophosphate:
[CH₃-cyc-(N⁺C₂H₂NCH)CH₂CH₃ PF₆⁻],
1,3-ethylmethylimidazolium tetrafluoroborate:
[CH₃-cyc-(N⁺C₂H₂NCH)CH₂CH₃ BF₄⁻],
- 15 1,2-dimethyl-3-propylimidazolium
bis(trifluoromethanesulfonyl)imide,
1,2-dimethyl-3-propylimidazolium
tris(trifluoromethanesulfonyl)methide,
1,2-dimethyl-3-propylimidazolium
- 20 trifluoromethanesulfonyl perfluorobutanesulfonylimide,
1-ethyl-3-methylimidazolium
cyanotrifluoromethanesulfonylamide,
1-ethyl-3-methylimidazolium
bis(cyano)trifluoromethanesulfonylmethide,
- 25 1-ethyl-3-methylimidazolium
trifluoromethanesulfonylperfluorobutanesulfonylimide,
octyldimethyl-2-hydroxyethylammonium
trifluoromethylsulfonylperfluorobutanesulfonylimide,
2-hydroxyethyltrimethyl
- 30 trifluoromethylsulfonylperfluorobutanesulfonylimide,
2-methoxyethyltrimethylammonium
bis(trifluoromethanesulfonyl)imide
octyldimethyl-2-hydroxyethylammonium
bis(cyano)trifluoromethanesulfonylmethide,

- trimethyl-2-acetoxyethylammonium
trifluoromethylsulfonylperfluorobutanesulfonylimide,
1-butylpyridinium
trifluoromethylsulfonylperfluorobutanesulfonylimide,
5 2-ethoxyethyltrimethylammonium
trifluoromethanesulfonate,
1-butyl-3-methylimidazolium perfluorobutanesulfonate,
perfluoro-1-ethyl-3-methylimidazolium
bis(trifluoromethanesulfonyl)imide,
10 1-ethyl-2-methylpyrazolium perfluorobutanesulfonate,
1-butyl-2-ethylpyrazolium trifluoromethanesulfonate,
N-ethylthiazolium bis(trifluoromethanesulfonyl)imide,
N-ethyloxazolium bis(trifluoromethanesulfonyl)imide,
and 1-butylpyrimidinium
15 perfluorobutanesulfonylbis(trifluoromethanesulfonyl)-
methide, 1,3-ethylmethylimidazolium
hexafluorophosphate, 1,3-ethylmethylimidazolium
tetrafluoroborate, and mixtures thereof.
- Preferred ionic salts include octyldimethyl-
- 20 2-hydroxyethylammonium
bis(trifluoromethylsulfonyl)imide:
[C₈H₁₇N⁺(CH₃)₂CH₂CH₂OH ⁻N(SO₂CF₃)₂],
octyldimethyl-2-hydroxyethylammonium
perfluorobutanesulfonate:
25 [C₈H₁₇N⁺(CH₃)₂CH₂CH₂OH ⁻OSO₂C₄F₉],
octyldimethyl-2-hydroxyethylammonium
trifluoromethanesulfonate:
[C₈H₁₇N⁺(CH₃)₂CH₂CH₂OH ⁻OSO₂CF₃],
octyldimethyl-2-hydroxyethylammonium
30 tris(trifluoromethanesulfonyl)methide:
[C₈H₁₇N⁺(CH₃)₂CH₂CH₂OH ⁻C(SO₂CF₃)₃],
trimethyl-2-acetoxyethylammonium
bis(trifluoromethylsulfonyl)imide:
[(CH₃)₃N⁺CH₂CH₂OC(O)CH₃ ⁻N(SO₂CF₃)₂],

- trimethyl-2-hydroxyethylammonium
bis(perfluorobutanesulfonyl)imide:
[$(\text{CH}_3)_3\text{N}^+\text{CH}_2\text{CH}_2\text{OH} \quad ^-\text{N}(\text{SO}_2\text{C}_4\text{F}_9)_2$],
- triethylammonium bis(perfluoroethanesulfonyl)imide:
5 [Et₃N⁺H⁻ N(SO₂C₂F₅)₂],
tetraethylammonium trifluoromethanesulfonate:
[CF₃SO₃⁻ ⁺NEt₄],
tetraethylammonium bis(trifluoromethanesulfonyl)imide:
[(CF₃SO₂)₂N⁻ ⁺NEt₄],
- 10 tetramethylammonium
tris(trifluoromethanesulfonyl)methide:
[(CH₃)₄N⁺ ⁻C(SO₂CF₃)₃],
tetrabutylammonium bis(trifluoromethanesulfonyl)imide:
[(C₄H₉)₄N⁺ ⁻N(SO₂CF₃)₂],
- 15 trimethyl-3-perfluorooctylsulfonamidopropylammonium
bis(trifluoromethanesulfonyl)imide:
[C₈F₁₇SO₂NH(CH₂)₃N⁺(CH₃)₃ ⁻N(SO₂CF₃)₂],
1-hexadecylpyridinium
bis(perfluoroethanesulfonyl)imide:
20 [n-C₁₆H₃₃-cyc-N⁺C₅H₅ ⁻N(SO₂C₂F₅)₂],
1-hexadecylpyridinium perfluorobutanesulfonate:
[n-C₁₆H₃₃-cyc-N⁺C₅H₅ ⁻OSO₂C₄F₉],
1-hexadecylpyridinium perfluorooctanesulfonate:
[n-C₁₆H₃₃-cyc-N⁺C₅H₅ ⁻OSO₂C₈F₁₇],
- 25 n-butylpyridinium bis(trifluoromethanesulfonyl)imide:
[n-C₄H₉-cyc-N⁺C₅H₅ ⁻N(SO₂CF₃)₂],
n-butylpyridinium perfluorobutanesulfonate:
[n-C₄H₉-cyc-N⁺C₅H₅ ⁻OSO₂C₄F₉],
1,3-ethylmethyylimidazolium
30 bis(trifluoromethanesulfonyl)imide:
[CH₃-cyc-(N⁺C₂H₂NCH)CH₂CH₃ ⁻N(SO₂CF₃)₂],
1,3-ethylmethyylimidazolium nonafluorobutanesulfonate:
[CH₃-cyc-(N⁺C₂H₂NCH)CH₂CH₃ ⁻OSO₂C₄F₉],

1,3-ethylmethylimidazolium trifluoromethanesulfonate:
[CH₃-cyc-(N⁺C₂H₂NCH)CH₂CH₃ ⁻OSO₂CF₃],
1,3-ethylmethylimidazolium tetrafluoroborate, and
mixtures thereof.

5 More preferred ionic salts include 2-
hydroxyethylammonium bis(trifluoromethylsulfonyl)imide,
octyldimethyl-2-hydroxyethylammonium
perfluorobutanesulfonate, octyldimethyl-2-
hydroxyethylammonium trifluoromethanesulfonate,
10 triethylammonium bis(perfluoroethanesulfonyl)imide,
tetraethylammonium trifluoromethanesulfonate,
trimethyl-3-perfluorooctylsulfonamidopropylammonium
bis(trifluoromethanesulfonyl)imide, 1,3-
ethylmethylimidazolium nonafluorobutanesulfonate, 1,3-
15 ethylmethylimidazolium
bis(trifluoromethanesulfonyl)imide, 1,3-
ethylmethylimidazolium trifluoromethanesulfonate, and
mixtures thereof.

 Most preferred ionic salts include 2-
20 hydroxyethylammonium bis
(trifluoromethylsulfonyl)imide, octyldimethyl-2-
hydroxyethylammonium trifluoromethanesulfonate,
triethylammonium bis(perfluoroethanesulfonyl)imide,
1,3-ethylmethylimidazolium nonafluorobutanesulfonate,
25 1,3-ethylmethylimidazolium
bis(trifluoromethanesulfonyl)imide, 1,3-
ethylmethylimidazolium trifluoromethanesulfonate, and
mixtures thereof, with further preferences being in
accordance with the general cation and anion
30 preferences set forth above.

 Insulating materials that are suitable for
topical treatment include materials that have
relatively low surface and bulk conductivity and that
are prone to static charge buildup. Such materials
35 include both synthetic and naturally-occurring polymers

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5 (or the reactive precursors thereof, for example, mono-
or multifunctional monomers or oligomers) that can be
either organic or inorganic in nature, as well as
ceramics, glasses, and ceramers (or the reactive
precursors thereof).

Suitable synthetic polymers (which can be
either thermoplastic or thermoset) include commodity
plastics such as, for example, poly(vinyl chloride),
polyethylenes (high density, low density, very low
10 density), polypropylene, and polystyrene; engineering
plastics such as, for example, polyesters (including,
for example, poly(ethylene terephthalate) and
poly(butylene terephthalate)), polyamides (aliphatic,
amorphous, aromatic), polycarbonates (for example,
15 aromatic polycarbonates such as those derived from
bisphenol A), polyoxymethylenes, polyacrylates and
polymethacrylates (for example, poly(methyl
methacrylate)), some modified polystyrenes (for
example, styrene-acrylonitrile (SAN) and acrylonitrile-
20 butadiene-styrene (ABS) copolymers), high-impact
polystyrenes (SB), fluoroplastics, and blends such as
poly(phenylene oxide)-polystyrene and polycarbonate-
ABS; high-performance plastics such as, for example,
liquid crystalline polymers (LCPs), polyetherketone
25 (PEEK), polysulfones, polyimides, and polyetherimides;
thermosets such as, for example, alkyd resins, phenolic
resins, amino resins (for example, melamine and urea
resins), epoxy resins, unsaturated polyesters
(including so-called vinyl esters), polyurethanes,
30 allyllics (for example, polymers derived from
allyldiglycolcarbonate), fluoroelastomers, and
polyacrylates; and the like and blends thereof.
Suitable naturally occurring polymers include
proteinaceous materials such as silk, wool, and

leather; and cellulosic materials such as cotton and wood.

Particularly useful insulating materials are thermoplastic polymers, including those described
5 above, as such polymers can be used in preparing the antistatic composition of the invention. Preferably, the thermoplastic polymers are melt processable at elevated temperatures, for example, above about 150°C (more preferably, above about 250°C; even more
10 preferably, above about 280°C; most preferably, above about 320°C). Preferred thermoplastic polymers include, for example, polypropylene, polyethylene, copolymers of ethylene and one or more alpha-olefins (for example, poly(ethylene-butene) and poly(ethylene-octene)),
15 polyesters, polyurethanes, polycarbonates, polyetherimides, polyimides, polyetherketones, polysulfones, polystyrenes, ABS copolymers, polyamides, fluoroelastomers, and blends thereof. More preferred are polypropylene, polyethylene, polyesters,
20 polyurethanes, polycarbonates, and blends thereof, with polypropylene, polycarbonates, polyesters, and blends thereof being most preferred.

The antistatic composition of the invention can generally be prepared by combining at least one
25 ionic salt (alone or in combination with other additives) and at least one thermoplastic polymer and then melt processing the resulting combination. Alternative processes for preparing an antistatic composition include, for example, (a) combining at
30 least one ionic salt (alone or in combination with other additives) and at least one thermosetting polymer or ceramer (or the reactive precursors thereof) and then allowing the resulting combination to cure, optionally with the application of heat or actinic
35 radiation; (b) applying a treatment composition

comprising at least one ionic salt to at least a portion of at least one surface of at least one insulating material; (c) dissolving at least one ionic salt and at least one insulating material in at least one solvent and then casting or coating the resulting solution and allowing evaporation of the solvent, optionally with the application of heat; and (d) combining at least one ionic salt (alone or in combination with other additives) and at least one monomer and then allowing polymerization of the monomer to occur, optionally in the presence of at least one solvent and optionally with the application of heat or actinic radiation.

To form a melt blend by melt processing, the ionic salt(s) can be, for example, intimately mixed with pelletized or powdered polymer and then melt processed by known methods such as, for example, molding, melt blowing, melt spinning, or melt extrusion. The salt(s) can be mixed directly with the polymer or they can be mixed with the polymer in the form of a "master batch" (concentrate) of the salt(s) in the polymer. If desired, an organic solution of the salt(s) can be mixed with powdered or pelletized polymer, followed by drying (to remove solvent) and then by melt processing. Alternatively, molten salt(s) can be injected into a molten polymer stream to form a blend immediately prior to, for example, extrusion into fibers or films or molding into articles.

After melt processing, an annealing step can be carried out to enhance the development of antistatic characteristics. In addition to, or in lieu of, such an annealing step, the melt processed combination (for example, in the form of a film or a fiber) can also be embossed between two heated rolls, one or both of which can be patterned. An annealing step typically is

conducted below the melt temperature of the polymer (for example, in the case of polyamide, at about 150-220°C for a period of about 30 seconds to about 5 minutes). In some cases, the presence of moisture can
5 improve the effectiveness of the ionic salt(s), although the presence of moisture is not necessary in order for antistatic characteristics to be obtained.

The ionic salt(s) can be added to thermoplastic polymer (or, alternatively, to other
10 insulating material) in an amount sufficient to achieve the desired antistatic properties for a particular application. This amount can be determined empirically and can be adjusted as necessary or desired to achieve the antistatic properties without compromising the
15 properties of the polymer (or other insulating material). Generally, the ionic salt(s) can be added in amounts ranging from about 0.1 to about 10 percent by weight (preferably, from about 0.5 to about 2 percent; more preferably, from about 0.75 to about 1.5
20 percent) based on the weight of polymer (or other insulating material).

In topical treatment of an insulating material, the ionic salt(s) can be employed alone or in the form of aqueous suspensions, emulsions, or
25 solutions, or as organic solvent solutions, in the topical treatment of the insulating material. Useful organic solvents include chlorinated hydrocarbons, alcohols (for example, isopropyl alcohol), esters, ketones (for example, methyl isobutyl ketone), and
30 mixtures thereof. Generally, the solvent solutions can contain from about 0.1 to about 50 percent, or even up to about 90 percent, by weight non-volatile solids (based on the total weight of the components). Aqueous suspensions, emulsions, or solutions are generally
35 preferred and generally can contain a non-volatile

solids content of about 0.1 to about 50 percent, preferably, about 1 to about 10 percent, by weight (based on the total weight of the components).

Preferably, however, topical treatment is carried out
5 by applying (to at least a portion of at least one surface of at least one insulating material) a topical treatment composition that consists essentially of at least one ionic salt that is liquid at the use or treatment temperature. Such a topical treatment
10 process involves the use of the neat liquid ionic salt, without added solvent, and is thus preferred from an environmental perspective over the use of organic solvent solutions of ionic salt(s).

The liquid ionic salt(s) (or suspensions,
15 emulsions, or solutions of liquid or solid ionic salt(s)) can be applied to an insulating material by standard methods such as, for example, spraying, padding, dipping, roll coating, brushing, or exhaustion (optionally followed by the drying of the treated
20 material to remove any remaining water or solvent). The material can be in the form of molded or blown articles, sheets, fibers (as such or in aggregated form, for example, yarn, toe, web, or roving, or in the form of fabricated textiles such as carpets), woven and
25 nonwoven fabrics, films, etc. If desired, the salt(s) can be co-applied with conventional fiber treating agents, for example, spin finishes or fiber lubricants.

The liquid ionic salts(s) (or suspensions, emulsions, or solutions of liquid or solid ionic
30 salt(s)) can be applied in an amount sufficient to achieve the desired antistatic properties for a particular application. This amount can be determined empirically and can be adjusted as necessary or desired to achieve the antistatic properties without
35 compromising the properties of the insulating material.

Any of a wide variety of constructions can be made from the antistatic composition of the invention, and such constructions will find utility in any application where some level of antistatic characteristic is required. For example, the antistatic composition of the invention can be used to prepare films and molded or blown articles, as well as fibers (for example, melt-blown or melt-spun fibers, including microfibers) that can be used to make woven and nonwoven fabrics. Such films, molded or blown articles, fibers, and fabrics exhibit antistatic characteristics under a variety of environmental conditions and can be used in a variety of applications.

For example, molded articles comprising the antistatic composition of the invention can be prepared by standard methods (for example, by high temperature injection molding) and are particularly useful as, for example, headlamp covers for automobiles, lenses (including eyeglass lenses), casings or circuit boards for electronic devices (for example, computers), screens for display devices, windows (for example, aircraft windows), and the like. Films comprising the antistatic composition of the invention can be made by any of the film making methods commonly employed in the art. Such films can be nonporous or porous (the latter including films that are mechanically perforated), with the presence and degree of porosity being selected according to the desired performance characteristics. The films can be used as, for example, photographic films, transparency films for use with overhead projectors, tape backings, substrates for coating, and the like.

Fibers comprising the composition of the invention can be used to make woven or nonwoven fabrics

that can be used, for example, in making medical fabrics, medical and industrial apparel, fabrics for use in making clothing, home furnishings such as rugs or carpets, and filter media such as chemical process filters or respirators. Nonwoven webs or fabrics can be prepared by processes used in the manufacture of either melt-blown or spunbonded webs. For example, a process similar to that described by Wentz in "Superfine Thermoplastic Fibers," Indus. Eng'g Chem. 48, 1342 (1956) or by Wentz et al. in "Manufacture of Superfine Organic Fibers," Naval Research Laboratories Report No. 4364 (1954) can be used. Multi-layer constructions made from nonwoven fabrics enjoy wide industrial and commercial utility, for example, as medical fabrics. The makeup of the constituent layers of such multi-layer constructions can be varied according to the desired end-use characteristics, and the constructions can comprise two or more layers of melt-blown and spunbonded webs in many useful combinations such as those described in U.S. Patent Nos. 5,145,727 (Potts et al.) and 5,149,576 (Potts et al.), the descriptions of which are incorporated herein by reference.

The ionic salts used in the antistatic composition of the invention can also find utility as additives to coatings (for example, polymer or ceramer coatings). Such coatings can be both antistatic and scratch-resistant and can be used in the photographic industry or as protective coatings for optical or magnetic recording media.

If desired, the antistatic composition of the invention can further contain one or more conventional additives commonly used in the art, for example, dyes, pigments, antioxidants, ultraviolet stabilizers, flame retardants, surfactants, plasticizers (for example,

polymers such as polybutylene), tackifiers, fillers, and mixtures thereof.

Objects and advantages of this invention are further illustrated by the following examples, but the particular materials and amounts thereof recited in these examples, as well as other conditions and details, should not be construed to unduly limit this invention.

10

GLOSSARY

HTS 905A - Larostat™ HTS 905A, $C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^-OSO_2CH_3$, available from BASF, Gurnee, Illinois.

HQ-115 - $LiN(SO_2CF_3)_2$ available from 3M, St. Paul, MN.

PBSF - Perfluorobutanesulfonyl fluoride, available from Sigma-Aldrich, Milwaukee, WI.

Lithium triflate - Lithium trifluoromethanesulfonate, available from Sigma-Aldrich, Milwaukee, WI.

FC-24 - Trifluoromethanesulfonic acid, available from 3M, St. Paul, MN.

FC-754 - Trimethyl-3-perfluorooctylsulfonamidopropylammonium chloride, available from 3M, St. Paul, MN.

Aliquat™ 336 - Methyltrioctylammonium chloride, available from Sigma-Aldrich, Milwaukee, WI, or from Henkel Corp., Ambler, PA.

FC-94 - Lithium perfluorooctanesulfonate, available from 3M, St. Paul, MN.

Cetylpyridinium chloride monohydrate - 1-Hexadecylpyridinium chloride, available from Research Organics, Cleveland, OH.

1,3-Ethylmethylimidazolium chloride -

Available from Sigma-Aldrich, Milwaukee, WI.

Silver triflate - Silver

trifluoromethanesulfonate, available from Sigma-

5 Aldrich, Milwaukee, WI.

AgBF₄ - Silver tetrafluoroborate, available

from Sigma-Aldrich, Milwaukee, WI.

NH₄PF₆ - Ammonium hexafluorophosphate,

available from Sigma-Aldrich, Milwaukee, WI.

10 **Acetylcholine chloride - CH₃CO₂CH₂CH₂N(CH₃)₃Cl,**

available from Research Organics, Cleveland, OH.

Choline chloride - HOCH₂CH₂N(CH₃)₃Cl, available

from Sigma-Aldrich, Milwaukee, WI.

PP3505 - ESCORENE™ PP3505 polypropylene,

15 having a 400 melt index flow rate, available from Exxon Chemical Co., Baytown, Texas.

PE6806 - ASPUN™ 6806 polyethylene, having a

melt flow index of 105 g/10 min (as measured by Test Method ASTM D-1238) and having a peak melting point of

20 124.8°C, available from Dow Chemical Co., Midland, Michigan.

PS440-200 - MORTHANE™ PS440-200 urethane,

available from Morton Thiokol Corp., Chicago, Illinois.

PET 65-1000 - polyethylene terephthalate

25 available from the 3M Company, Decatur, AL.

LQ-3147 - Makrolon® LQ-3147 polycarbonate

available from Bayer Corp., Pittsburgh, PA.

Mellinex 617 - Melamine primed polyethylene

terephthalate film (0.177 mm thick), available from

30 DuPont, Hopewell, VA.

TEST METHODS

Test Method I - Melting Point Determination

The melting points of salts were determined by differential scanning calorimetry (DSC) using a 20°C per minute temperature ramp. The peak maximum of the melt transition was taken as the melting point (T_m). Where multiple melt transitions were observed, the peak associated with largest area melt transition was taken as the melting point.

10 **Test Method II - Onset of Thermal Decomposition Determination**

The onset of thermal decomposition of each salt was determined by thermal gravimetric analysis (TGA) under an inert nitrogen atmosphere using a 10°C per minute temperature ramp. The value of the onset temperature was determined by finding the intersection of the extrapolated tangent at the baseline preceding onset and the extrapolated tangent at the inflection point associated with the step change in sample weight.

20

Test Method III - Static Charge Dissipation Test

The static charge dissipation characteristics of nonwoven fabrics, films, and molded sheets were determined with this method. The test materials were cut into 9 cm by 12 cm samples and conditioned at relative humidities (RH) of about 10 percent, 25 percent, and 50 percent for at least 12 hours. The materials were tested at temperatures that ranged from 22-25°C. The static charge dissipation time was measured according to Federal Test Method Standard 10113, Method 4046, "Antistatic Properties of Materials", using an ETS Model 406C Static Decay Test Unit (manufactured by Electro-Tech Systems, Inc., Glenside, PA). This apparatus induces an initial static charge (Average

Induced Electrostatic Charge) on the surface of the flat test material by using high voltage (5000 volts), and a fieldmeter allows observation of the decay time of the surface voltage from 5000 volts (or whatever the induced electrostatic charge was) to 10 percent of the initial induced charge. This is the static charge dissipation time. The lower the static charge dissipation time, the better the antistatic properties are of the test material. All reported values of the static charge dissipation times in this invention are averages (Average Static Decay Rate) over at least 3 separate determinations. Values reported as > 60 sec indicate that the material tested has an initial static charge which cannot be removed by surface conduction and is not antistatic.

Test Method IV - Surface Resistivity Test

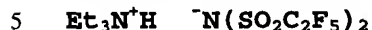
This test was conducted according to the procedure of ASTM Standard D-257, "D.C. Resistance or Conductance of Insulating Materials". The surface resistivity was measured under the conditions of this test method using an ETS Model 872 Wide Range Resistance Meter fitted with a Model 803B probe (Electro-Tech Systems, Inc., Glenside, PA). This apparatus applies an external voltage of 100 volts across two concentric ring electrodes contacting the flat test material, and provides surface resistivity readings in ohm/square units.

In the following where weight percent or parts by weight are indicated, these are basis on the weight of the entire composition unless indicated otherwise.

PREPARATION OF COMPOUNDS

Compound 1

**Synthesis of Triethylammonium
bis(perfluoroethanesulfonyl)imide,**



The title compound was prepared according to the method described in US 5,874,616, Example 3, except that the procedure was terminated once the methylene chloride solvent was evaporated. The product was
10 characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 2.

15 **Compound 2**

**Synthesis of Tetraethylammonium
trifluoromethanesulfonate, $\text{CF}_3\text{SO}_3^- \text{NEt}_4^+$**

In a 2L flask, 300 g of $\text{CF}_3\text{SO}_3\text{H}$ (FC-24) was charged. The acid was neutralized by slow addition of
20 about 800g Et_4NOH aqueous solution (35%) until the pH reached about 6. A white solid (560g) was obtained after drying by rotary evaporation, then under high vacuum. The solid was re-crystallized from chloroform-heptane to give 520g pure product. The product was also
25 characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 2.

30 **Compound 3**

**Synthesis of Tetraethylammonium
bis(trifluoromethanesulfonyl)imide, $(\text{CF}_3\text{SO}_2)_2\text{N}^- \text{NEt}_4^+$ in
Water- CH_2Cl_2 Mixed Solvent**

In a 1L flask, 50 g of $(\text{CF}_3\text{SO}_2)_2\text{N}^- \text{Li}^+$ (HQ-115) was dissolved in 50 g of deionized water. The solution was combined with 89 g of 35% Et_4NOH aqueous solution under N_2 . Solid precipitated during the addition, which was dissolved by the addition of 50 g CH_2Cl_2 . The bottom organic layer was isolated. The aqueous solution was extracted with another 50 g of CH_2Cl_2 . The combined organic solution was washed with water (2 x 25 mL), and volatiles were removed by rotary evaporation. Re-crystallization of the crude product from $\text{CH}_3\text{OH}-\text{H}_2\text{O}$ gave 70g of white solid after full vacuum drying. The product was characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 2.

Compound 4

Synthesis of Tetramethylammonium

tris(trifluoromethanesulfonyl)methide, $(\text{CH}_3)_4\text{N}^+$

20 **$\text{C}(\text{SO}_2\text{CF}_3)_3$**

The title compound was prepared according to the method of US 5,554,664, Example 18, except that the procedure was terminated after line 55. The product was characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 2.

Compound 5

30 **Synthesis of Tetrabutylammonium**

bis(trifluoromethanesulfonyl)imide,

$(\text{C}_4\text{H}_9)_4\text{N}^+ \text{N}^-(\text{SO}_2\text{CF}_3)_2$

The title compound was prepared by reacting $(\text{C}_4\text{H}_9)_4\text{N}^+\text{Br}^-$ (Sigma-Aldrich, Milwaukee, WI) with

approximately a 10% molar excess of $\text{Li}^+ \text{N}(\text{SO}_2\text{CF}_3)_2$ (HQ-115) according to the procedure described in Example 18. The product was characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 2.

Compound 6

Synthesis of 1-Hexadecylpyridinium

Bis(perfluoroethanesulfonyl)imide,

$n\text{-C}_{16}\text{H}_{33}\text{-cyc-N}^+\text{C}_5\text{H}_5 \text{N}(\text{SO}_2\text{CF}_3)_2$

The title compound was prepared according to the method of Example 14, except that 85.1g of $\text{Li}^+ \text{N}(\text{SO}_2\text{CF}_3)_2$ (HQ-115) was employed as the anion precursor. The product was characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 3.

Compound 7

Synthesis of 1-Hexadecylpyridinium

Perfluorobutanesulfonate,

$n\text{-C}_{16}\text{H}_{33}\text{-cyc-N}^+\text{C}_5\text{H}_5 \text{OSO}_2\text{C}_4\text{F}_9$

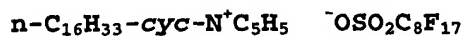
Cetylpyridinium chloride monohydrate (75g) was dissolved in 800ml water with gentle heating and magnetic stirring. To this solution was added 67.3g of $\text{Li}^+ \text{OSO}_2\text{C}_4\text{F}_9$ (prepared by hydrolysis of $\text{C}_4\text{F}_9\text{SO}_2\text{F}$ [PBSF] with LiOH) dissolved in 600mL of water with stirring. Product precipitated immediately and was isolated by suction filtration. The product was washed with copious amounts of water and then dried initially by suction and then in vacuo at 10^{-2} Torr, 40°C . The product was characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d)

according to Test Method II. Results are shown in Table 3.

Compound 8

5 **Synthesis of 1-Hexadecylpyridinium**

Perfluorooctanesulfonate,



The title compound was prepared according to the method of Example 14, except that 111.3g of $\text{Li}^+ \text{ } ^-\text{OSO}_2\text{C}_8\text{F}_{17}$ (FC-94) was employed as the anion precursor. The product was characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 3.

15

Compound 9

Synthesis of n-Butylpyridinium

Bis(trifluoromethanesulfonyl)imide,



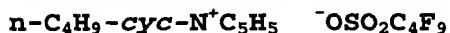
20 A solution of 50g $\text{Li}^+ \text{ } ^-\text{N}(\text{SO}_2\text{CF}_3)_2$ (HQ-115) (287 g/mol, 0.174 mol) and 100 ml DI water was prepared. Another solution of 30g butylpyridinium chloride (171.6 g/mol, 0.174) and 100 ml DI water was prepared. The two solutions were added to a separatory funnel along
25 with 200 ml methylene chloride. The mixture was thoroughly shaken, and the phases were allowed to separate. The organic phase was isolated and washed with 3 x 200 ml DI water. The organic layer was then concentrated by reduced pressure distillation on a
30 rotary evaporator. The resulting yellow oil was vacuum dried at 120C overnight to afford 70g product (97% yield). The product was characterized for melting point (T_m) according to Test Method I and for onset of

thermal decomposition (T_d) according to Test Method II. Results are shown in Table 3.

Compound 10

5 Synthesis of n-Butylpyridinium

Perfluorobutanesulfonate,



A solution of 20g butylpyridinium chloride (171.6 g/mol, 0.116 mol) was made with 100 ml DI water. A similar solution was prepared using 35.7g $\text{Li}^+\text{OSO}_2\text{C}_4\text{F}_9$ (prepared by hydrolysis of $\text{C}_4\text{F}_9\text{SO}_2\text{F}$ [PBSF] with LiOH) (306 g/mol, 0.116 mol) and 100 ml water. The two solutions were added to a separatory funnel along with 200 ml methylene chloride. The mixture was thoroughly shaken, and the phases were allowed to separate. The organic phase was isolated and washed with 200 ml DI water. The mixture was slow to separate, consequently further washings were not done. The organic layer was concentrated by reduced pressure distillation on a rotary evaporator. It was then dried under vacuum at 130C overnight. The isolated yellow oil weighed 44g (87% yield). The product was characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 3.

Compound 11

Synthesis of 1,3-Ethylmethylimidazolium

Bis(trifluoromethanesulfonyl)imide,



1,3-Ethylmethylimidazolium chloride (50.0g) and $\text{LiN(SO}_2\text{CF}_3)_2$ (HQ-115) (102.8g) were combined in 500mL of water with magnetic stirring. A nonmiscible light yellow oil of low viscosity separated as a lower

liquid phase. The mixture was transferred to a separatory funnel combined with 500mL of CH₂Cl₂ and the workup conducted essentially as described in Example 1. After vacuum stripping all volatiles, a total of 112.2g (84% yield) of light yellow oil of high purity was obtained, which was identified as the title compound by ¹H and ¹⁹F NMR. The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 4.

Compound 12

Synthesis of 1,3-Ethylmethylimidazolium

Nonafluorobutanesulfonate,

CH₃-cyc-(N⁺C₂H₂NCH)CH₂CH₃ ⁻OSO₂C₄F₉,
 1,3-Ethylmethylimidazolium chloride (49.1g) and LiOSO₂C₄F₉ (107.6g, prepared by hydrolysis of C₄F₉SO₂F with LiOH) were combined in 500mL of water with magnetic stirring. A homogeneous aqueous solution was formed, which was transferred to a separatory funnel, combined with 500mL of CH₂Cl₂ and worked up according to the procedure in Example 1. After vacuum stripping all volatiles, a total of 65.0g (47% yield) of light yellow oil of high purity was obtained, which was identified as the title compound by ¹H and ¹⁹F NMR. The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 4.

30

Compound 13

Synthesis of 1,3-Ethylmethylimidazolium

trifluoromethanesulfonate,

CH₃-cyc-(N⁺C₂H₂NCH)CH₂CH₃ ⁻OSO₂CF₃

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1,3-Ethylmethylimidazolium chloride (29g, 0.199 mole) was dissolved in 100 ml of water and added to solution of 50g silver triflate (0.195 mol) in 200g water with stirring. The silver chloride precipitate was removed by filtration, and the solids were washed with 100ml of deionized water. The filtrate was concentrated on a rotary evaporator and further dried at 75°C overnight to provide 47.5g of a light green oil that was characterized by ^1H and ^{19}F NMR. The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 4.

Compound 14

Synthesis of 1,3-Ethylmethylimidazolium

Tetrafluoroborate,



Separate solutions of 49.6g AgBF_4 (194.68g/mol, 0.255 mol) in 200 ml distilled water, and 37.35g 1,3-Ethylmethylimidazolium chloride (146.62 g/mol, 0.255 mol) in 200 ml distilled water were prepared. The two solutions were mixed together, instantly forming a white precipitate. The solution was allowed to settle, followed by filtration through a D-frit. The filtrate was concentrated, but not to dryness and allowed to stand at room temperature overnight. The next morning a black precipitate was observed to have fallen out of solution. The solution was passed through filter paper to removed the small amount of solid. The remaining water was removed by reduced pressure distillation on a rotary evaporator. The remaining oil was dissolved in 200 ml acetonitrile. More insoluble black precipitate was formed and was filtered out of the solution. The yellow filtrate was

concentrated on the rotary evaporator, and the resulting oil was dried overnight under vacuum at 75C. The isolated weight of product was 40g (79% yield). The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 4.

Compound 15

10 **Synthesis of 1,3-Ethylmethylimidazolium
Hexafluorophosphate,
 $\text{CH}_3\text{-cyc-(N}^+\text{C}_2\text{H}_2\text{NCH)}\text{CH}_2\text{CH}_3 \text{ PF}_6^-$**

15 A solution of 500 ml acetonitrile and 73.1g 1,3-Ethylmethylimidazolium chloride (146.6 g/mol, 0.498 mol) was prepared in a 1L flask. Another solution of 250 ml acetonitrile and 81.1g NH_4PF_6 (163 g/mol, 0.498 mol) was similarly prepared and added to the former solution. A white precipitate instantly formed on mixing of the two solutions. The flask was chilled to 20 near 0°C for 1 hour followed by filtration through high purity Celite using a D-frit. The solvent was removed by reduced pressure distillation on a rotary evaporator. The ionic salt was dried under vacuum at 75C overnight. The isolated weight of product was 114g 25 (89% yield). The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 4.

30

EXAMPLES

Example 1

**Synthesis of Octyldimethyl-2-hydroxyethylammonium
bis(trifluoromethylsulfonyl)imide, $\text{C}_8\text{H}_{17}\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{CH}_2\text{OH}$
 $\text{N}(\text{SO}_2\text{CF}_3)_2$**

A 19.2g sample of $C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^-OSO_2CH_3$ (HTS 905A) was combined with 15.7g $LiN(SO_2CF_3)_2$ (HQ-115) in 120mL of water. After agitating the mixture, a clear, nonmiscible oil separated as a lower liquid phase. The mixture was transferred to a separatory funnel and 125mL of methylene chloride was added. The mixture was shaken vigorously and allowed to phase separate. The lower organic phase was isolated and washed with two additional 125mL portions of water. The washed methylene chloride phase was isolated and over anhydrous aluminum oxide beads, filtered by suction and vacuum stripped at $30-100^\circ C$, $20 - 10^{-3}$ Torr to remove all volatiles. A colorless oil (22.6g, 85% yield) of high purity was obtained, which was identified as the title compound by 1H , ^{13}C and ^{19}F NMR. The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 1.

Example 2

Synthesis of Octyldimethyl-2-hydroxyethylammonium perfluorobutanesulfonate, $C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^-OSO_2C_4F_9$

A 118.5g (0.399 mol) sample of $C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^-OSO_2CH_3$ (HTS 905A) was dissolved in about 250 ml of water and 123.9g (0.399 mol) of $LiOSO_2C_4F_9$ (prepared by hydrolysis of $C_4F_9SO_2F$ [PBSF] with $LiOH$) was dissolved in about 100 ml of water. The two solutions were added to a separatory funnel and the mixture was shaken vigorously. Next 200ml of methylene chloride was added to the funnel and the contents were shaken and allowed to phase separate. The lower methylene chloride layer was washed twice with about 200 ml of water and concentrated on a rotary evaporator at about $85^\circ C$ for about 45 min to yield an off-white

solid, which was characterized by ^1H and ^{13}C nuclear magnetic resonance spectroscopy (NMR). The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 1.

Example 3

Synthesis of Octyldimethyl-2-hydroxyethylammonium

10 **trifluoromethanesulfonate, $\text{C}_8\text{H}_{17}\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{CH}_2\text{OH}^- \text{OSO}_2\text{CF}_3$**

Into 30g of acetonitrile in a 125 ml Erlenmeyer flask was dissolved with heating 29.7g (0.1 mole) HTS-905A ($\text{C}_8\text{H}_{17}\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{CH}_2\text{OH}^- \text{O}_3\text{SCH}_3$) and then cooled in an ice bath for 10 minutes. In another 125ml
15 Erlenmeyer flask was dissolved with heating 15.6g (0.1mole) lithium triflate into 30 ml of acetonitrile. Next the lithium triflate solution was added over about 1 min to the stirred, cooled HTS-905A solution with generation of a white precipitate. About 2 ml of
20 acetonitrile was used to rinse the Erlenmeyer flask that held the lithium triflate solution, and this was also added to the HTS-905A solution. The reaction mixture was allowed to stir for about 10 minutes and was then vacuum filtered through a pad of Celite on a
25 125ml Buchner funnel with a C porosity frit. The reaction flask and Celite pad were washed with an additional 30 g of ice-cold acetonitrile. The filtrate was concentrated on a rotary evaporator at about 50 mm Hg with a bath temperature of about 85°C for about 45
30 min to yield 24.5g of a clear solid, which was characterize by ^1H and ^{13}C NMR. The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in
35 Table 1.

Exempl 4

**Synthesis of Octyldimethyl-2-hydroxyethylammonium
tris(trifluoromethanesulfonyl)methide,**

5 **$C_8H_{17}N^+(CH_3)_2CH_2CH_2OH \ ^-C(SO_2CF_3)_3$**

A 20.0g sample of $C_8H_{17}N^+(CH_3)_2CH_2CH_2OH \ ^-OSO_2CH_3$
(HTS 905) was combined with 29.6g $HC(SO_2CF_3)_3$ (prepared
as described in Example 1 of U.S. Pat. No. 5,554,664)
in 250mL of water. After agitating the mixture, a
10 clear, viscous, pale yellow, nonmiscible oil separated
as a lower liquid phase. The mixture was transferred to
a separatory funnel, combined with 300mL of methylene
chloride, and worked up according to the procedure in
Example 1. After vacuum stripping all volatiles, a
15 total of 29.0g (79% yield) of pale yellow oil was
obtained, which was identified as the title compound by
 1H and ^{19}F NMR. Estimated purity from the NMR analysis
was greater than 90 weight %, the major impurity being
the corresponding $^-C(SO_2CF_3)_2(SO_2F)$ salt. The product was
20 also characterized for melting point (T_m) according to
Test Method I and for onset of thermal decomposition
(T_d) according to Test Method II. Results are shown in
Table 1.

25 **Example 5**

**Synthesis of Trimethyl-2-acetoxyethylammonium
bis(trifluoromethylsulfonyl)imide, $(CH_3)_3N^+CH_2CH_2OC(O)CH_3$
 $^-N(SO_2CF_3)_2$**

Acetylcholine chloride (98g, Research
30 Organics, Cleveland, OH) and $LiN(SO_2CF_3)_2$ (HQ-115)
(165.8g) were combined in 600mL of water with magnetic
stirring. A viscous, nonmiscible oil separated as a
lower liquid phase. The reaction mixture was worked up
essentially as described in Example 1, except that the
35 ionic liquid product was not completely miscible with

methylene chloride, forming 3 separate liquid phases in the presence of water. The lower ionic liquid phase and the middle CH₂Cl₂ phase were both carried through the workup. After vacuum stripping all volatiles, a total of 179.1g (77% yield) of colorless oil of high purity was obtained, which was identified as the title compound by ¹H, ¹³C and ¹⁹F NMR. The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 1.

Example 6

Synthesis of Trimethyl-2-hydroxyethylammonium

bis(perfluorobutanesulfonyl)imide, (CH₃)₃N⁺CH₂CH₂OH⁻ N(SO₂C₄F₉)₂

Choline chloride (37.34g, Aldrich) and LiN(SO₂C₄F₉)₂ (142.7g, prepared according to Example 4 in U.S. Pat. No. 5,874,616) were combined in 400mL of water with magnetic stirring. A viscous, nonmiscible oil separated as a lower liquid phase. The mixture was transferred to a separatory funnel and 110mL of diethyl ether were added. The mixture was shaken vigorously and allowed to phase separate. The lower organic phase was isolated and washed with two additional 400mL portions of water. The washed ether phase was isolated and vacuum stripped at 30-100°C , 20 - 10⁻³ Torr to remove all volatiles. A colorless oil (155.3g, 93% yield) of high purity was obtained, which was identified as the title compound by ¹H, ¹³C and ¹⁹F NMR. The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 1.

Comparative Example C1

Larostat™ HTS 905A,
octyldimethylhydroxyethylammonium methanesulfonate
($C_8H_{17}N^+(CH_3)_2C_2H_4OH^-OSO_2CH_3$) was also characterized for
5 melting point (T_m) according to Test Method I and for
onset of thermal decomposition (T_d) according to Test
Method II. Results are shown in Table 1.

Table 1. Melting Point (T_m) and Onset of Thermal
10 Decomposition (T_d) Values Found for Compounds
Synthesized in Examples 1-6.

Example	Compound	T_m (°C)	T_d (°C)
1	$C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^-N(SO_2CF_3)_2$	None detected	409
2	$C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^-OSO_2C_4F_9$	147	374
3	$C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^-OSO_2CF_3$	-26	370
4	$C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^-C(SO_2CF_3)_3$	None detected	387
5	$(CH_3)_3N^+CH_2CH_2OC(O)CH_3^-N(SO_2CF_3)_2$	24	361
6	$(CH_3)_3N^+CH_2CH_2OH^-N(SO_2C_4F_9)_2$	32	402
C1	$C_8H_{17}N^+(CH_3)_2C_2H_4OH^-OSO_2CH_3$	About 30	289

The results in Table 1 show that among
antistat having the same cation, those containing
15 weakly coordinating fluoroorganic anions (Examples 1-4)
exhibit greatly increased thermal stability over that
of Comparative Example C1 having a more strongly
coordinating anion. All examples of the invention show
good thermal stability.

20

Example 7

Synthesis of Trimethyl-3-
perfluorooctylsulfonamidopropylammonium
bis(trifluoromethanesulfonyl)imide,

25 $C_8F_{17}SO_2NH(CH_2)_3N^+(CH_3)_3^-N(SO_2CF_3)_2$

In a 4.0L separatory funnel was combined 800mL water, 400g of 50% FC-754 ($C_8F_{17}SO_2NH(CH_2)_3N^+(CH_3)_3Cl^-$), 90.4g $Li^+ N(SO_2CF_3)_2$ (HQ-115) and 700mL methyl-t-butyl ether (MTBE). The mixture was agitated and the upper and lower liquid phases allowed to separate overnight. The two liquid phases were isolated and the water phase was extracted with a fresh 500mL portion of MTBE. The ether phases were combined and extracted with a fresh 700mL portion of water. The isolated ether phase was dried over molecular sieves, filtered through paper, and the solvent was evaporated to dryness in a vacuum oven at 95°C and 300-400 Torr providing 267.3g of the title compound (96% Yield). The solid product was characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 2.

Comparative Example C2

Aliquat™ 336, methyltrioctylammonium chloride ($(C_8H_{17})_3N^+(CH_3)Cl^-$) was liquid at room temperature and was also characterized for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 2. Because of its low thermal decomposition temperature, this compound could not be incorporated into melt-blown fibers.

Table 2. Melting Point (T_m) and Onset of Thermal Decomposition (T_d) Values Found for Comparative Example C2 and Compounds 1-6.

Example or Compound No.	Compound	T _m (°C)	T _d (°C)
Compound 1	Et ₃ N ⁺ H ⁻ N(SO ₂ C ₂ F ₅) ₂	-10	351
Compound 2	CF ₃ SO ₃ ⁻ ⁺ NEt ₄	133	371
Compound 3	(CF ₃ SO ₂) ₂ N ⁻ ⁺ NEt ₄	8	426
Compound 4	(CH ₃) ₄ N ⁺ ⁻ C(SO ₂ CF ₃) ₃	148	422
Compound 5	(C ₄ H ₉) ₄ N ⁺ ⁻ N(SO ₂ CF ₃) ₂	93	401
7	C ₈ F ₁₇ SO ₂ NH(CH ₂) ₃ N ⁺ (CH ₃) ₃ ⁻ N(SO ₂ CF ₃) ₂	121	365
C2	(C ₈ H ₁₇) ₃ N ⁺ (CH ₃) Cl ⁻	< 28	177

The data in Table 2 shows that Example 7 and the compounds useful in the invention having the weakly coordinating fluoroorganic anions show much greater thermal stability than Comparative Example C2 having the more strongly coordinating chloride anion.

Comparative Example C3

Synthesis of 1-Hexadecylpyridinium p-Toluenesulfonate,

10 n-C₁₆H₃₃-cyc-N⁺C₅H₅ ⁻OSO₂C₆H₄-p-CH₃

The title compound was prepared according to the method of Example 14, except that 100g of cetylpyridinium chloride monohydrate was reacted with 55g Na⁺ ⁻OSO₂C₆H₄-p-CH₃ (Sigma-Aldrich, Milwaukee, WI).

15 The product was also characterized for melting point (T_m) according to Test Method I and for onset of thermal decomposition (T_d) according to Test Method II. Results are shown in Table 3.

20 Table 3. Melting Point (T_m) and Onset of Thermal Decomposition (T_d) Values Found for Comparative Example C3 and Compounds 7-11.

Example or Compound No.	Compound	T _m (°C)	T _d (°C)
Compound 6	n-C ₁₆ H ₃₃ -cyc-N ⁺ C ₅ H ₅ ⁻ N(SO ₂ C ₂ F ₅) ₂	34	396
Compound 7	n-C ₁₆ H ₃₃ -cyc-N ⁺ C ₅ H ₅ ⁻ OSO ₂ C ₄ F ₉	95	357
Compound 8	n-C ₁₆ H ₃₃ -cyc-N ⁺ C ₅ H ₅ ⁻ OSO ₂ C ₈ F ₁₇	93	364
Compound 9	n-C ₄ H ₉ -cyc-N ⁺ C ₅ H ₅ ⁻ N(SO ₂ CF ₃) ₂	33	430
Compound 10	n-C ₄ H ₉ -cyc-N ⁺ C ₅ H ₅ ⁻ OSO ₂ C ₄ F ₉	63	391
C3	n-C ₁₆ H ₃₃ -cyc-N ⁺ C ₅ H ₅ ⁻ OSO ₂ C ₆ H ₄ -p-CH ₃	138	310

Table 3 shows that the pyridinium compounds useful in the invention having the weakly coordinating fluoroorganic anions have greater thermal stability than Comparative Example C3 having the more strongly coordinating anion.

Table 4. Melting point (T_m) and Onset of Thermal Decomposition (T_d) Values Found for Compounds 11-15.

Compound No.	Compound	T _m (°C)	T _d (°C)
11	CH ₃ -cyc-(N ⁺ C ₂ H ₂ NCH)CH ₂ CH ₃ ⁻ N(SO ₂ CF ₃) ₂	-18	450
12	CH ₃ -cyc-(N ⁺ C ₂ H ₂ NCH)CH ₂ CH ₃ ⁻ OSO ₂ C ₄ F ₉	18	410
13	CH ₃ -cyc-(N ⁺ C ₂ H ₂ NCH)CH ₂ CH ₃ ⁻ OSO ₂ CF ₃	-16	429
14	CH ₃ -cyc-(N ⁺ C ₂ H ₂ NCH)CH ₂ CH ₃ BF ₄ ⁻	7	420
15	CH ₃ -cyc-(N ⁺ C ₂ H ₂ NCH)CH ₂ CH ₃ PF ₆ ⁻	70	490

The data of Table 4 show that the imidazolium compounds useful in the invention having the weakly coordinating fluoroorganic anions all have excellent thermal stability, with T_d all greater than 400°C.

Examples 23-46 and Comparative Examples C4-C7

The compounds of Examples 1- 7, Compounds 1-15, and Comparative Examples C1-C3 were incorporated into polypropylene melt blown fibers, which were processed into nonwoven fabrics according to the melt-

blown extrusion procedure described in U.S. Pat. No.
5,300,357, column 10, which is herein incorporated by
reference. For comparison, polypropylene melt blown
fibers without these compounds were processed into
5 nonwoven fabrics as well. The extruder used was a
Brabender 42 mm conical twin screw extruder, with
maximum extrusion temperature of 270-280°C and distance
to the collector of 12 inches (30 cm).

The compound and Escorene™ PP3505
10 polypropylene were mixed by blending in a paperboard
container using a mixer head affixed to a hand drill
for about one minute until a visually homogeneous
mixture is obtained. The compound was dispersed in the
molten polypropylene by mixing in the melt extrusion
15 apparatus just prior to melt blowing. Except as noted,
the weight percent of the compound in the polypropylene
was about 1%.

The process condition for each mixture was
the same, including the melt blowing die construction
20 used to blow the microfiber web, the basis weight of
the web ($50 \pm 5 \text{ g/m}^2$) and the diameter of the microfibers
(5-18 micrometers). Unless otherwise stated, the
extrusion temperature was 270-280°C, the primary air
temperature was 270°C, the pressure was 124 kPa (18
25 psi), with a 0.076 cm air gap width, and the polymer
throughput rate was about 180 g/hr/cm.

The resulting melt blown polypropylene fabric
made with and without the compounds of Examples 1-7,
compounds 1-15, and Comparative Examples C1-C3 were
30 evaluated for antistatic performance using Test Method
III - Static Charge Dissipation Test. The results are
shown in Table 5.

Table 5. Static Charge Dissipation of Escorene™
35 PP3505 Polypropylene With and Without Ionic Antistat

Compounds at 10, 25, and 50 Percent Relative Humidity
(RH) .

10032946 122304

TABLE 96E00T

Example No.	Antistat Compound	Aver. Induced Electrostatic Charge (Volts)			Aver. Static Decay Rate (sec)		
		10% RH	25% RH	50% RH	10% RH	25% RH	50% RH
C4	None	4270	5000+	5000+	60+	60	60
			1750	1750		>10	>10
			5000	5000		>10	>10
C5	$C_8H_{17}N^+(CH_3)_2C_2H_4OH^-$ OSO_2CH_3	5000+	3393	5000+	0.86	0.14	0.03
23	$C_8H_{17}N^+(CH_3)_2C_2H_4OH^-$ $N(SO_2CF_3)_2$	5000+	5000+	5000+	0.14	0.19	0.63
24	$C_8H_{17}N^+(CH_3)_2C_2H_4OH^-$ $N(SO_2CF_3)_2$ (0.5% by wt.)	Not run	5000	5000	Not run	>10	0.95
25	$C_8H_{17}N^+(CH_3)_2C_2H_4OH^-$ $OSO_2C_4F_9$	5000	5000	5000	0.90	0.02	0.02
26	$C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^-$ OSO_2CF_3	5000	5000	5000	4.17	0.09	0.03
27	$C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^-$ $C(SO_2CF_3)_3$	Not run	Not run	5000	Not run	Not run	>10
28	$CH_3COOCH_2CH_2N^+(CH_3)_3^-$ $N(SO_2CF_3)_2$	Not run	5000+	5000+	Not run	>60	>60
29	$HOCH_2CH_2N^+(CH_3)_3^-$ $N(O_2SC_4F_9)_2$	Not run	4850	5000+	Not run	56	>60
C6	$(C_8H_{17})_3N^+(CH_3)Cl^-$	NR*	NR*	NR*	NR*	NR*	NR*
30	$Et_3NH^+ - N(SO_2C_2F_5)_2^-$	5000	5000	5000	0.03	0.03	0.02
31	$CF_3SO_3^- ^+NEt_4$	Not	Not	5000	Not	Not	>10

TABLE "STEE001"

Example No.	Antistat Compound	Aver. Induced Electrostatic Charge (Volts)			Aver. Static Decay Rate (sec)		
		10% RH	25% RH	50% RH	10% RH	25% RH	50% RH
32	CF ₃ SO ₃ ⁻ ⁺ NEt ₄ (2% by wt.)	5000	run	5000	run	0.38	0.51
33	(CF ₃ SO ₂) ₂ N ⁻ ⁺ NEt ₄	Not run	Not run	5000	Not run	Not run	>10
34	(CH ₃) ₄ N ⁺ ⁻ C(SO ₂ CF ₃) ₃	Not run	4400	3350	Not run	60	60
35	Bu ₄ N ⁺ ⁻ N(SO ₂ CF ₃) ₂	Not run	3700	3600	Not run	60	60
36	C ₈ F ₁₇ SO ₂ NH(CH ₂) ₃ N ⁺ (CH ₃) ₃ ⁻ N(SO ₂ CF ₃) ₂	Not run	2200	1600	Not run	0.01	0.01
C7	n-C ₁₆ H ₃₃ N ⁺ C ₅ H ₅ ⁻ OSO ₂ C ₆ H ₄ -p-CH ₃	Not run	4900	4900	Not run	52	52
37	n-C ₁₆ H ₃₃ N ⁺ C ₅ H ₅ ⁻ N(SO ₂ C ₂ F ₅) ₂	4800	4700	5000+	34	8.9	45
38	n-C ₁₆ H ₃₃ N ⁺ C ₅ H ₅ ⁻ OSO ₂ C ₄ F ₉	Not run	5000+	5000+	Not run	56	1.01
39	n-C ₁₆ H ₃₃ N ⁺ C ₅ H ₅ ⁻ OSO ₂ C ₈ F ₁₇	Not run	3600	5000+	Not run	>60	3.98
40	n-C ₄ H ₉ N ⁺ C ₅ H ₅ ⁻ N(O ₂ SCF ₃) ₂	Not run	5000+	4300	Not run	49	0.06

TABLE "A" CONT

Example No.	Antistat Compound	Aver. Induced Electrostatic Charge (Volts)			Aver. Static Decay Rate (sec)		
		10% RH	25% RH	50% RH	10% RH	25% RH	50% RH
41	$n-C_4H_9N^+C_5H_5^-O_3SC_4F_9$	Not run	5000+	5000+	Not run	>60	0.46
42	$CH_3-cyc-(N^+C_2H_2NCH)CH_2CH_3-N(SO_2CF_3)_2$	4125	3900	3350	60+	0.01	53
43	$CH_3-cyc-(N^+C_2H_2NCH)CH_2CH_3-OSO_2C_4F_9$	5000+	5000+	5000+	0.02	20	0.03
44	$CH_3-cyc-(N^+C_2H_2NCH)CH_2CH_3-OSO_2CF_3$	Not run	2500	2500	Not run	>10	0.01
45	$CH_3-cyc-(N^+C_2H_2NCH)CH_2CH_3-BF_4^-$	4500	4950	4250	>60	>60	>60
46	$CH_3-cyc-(N^+C_2H_2NCH)CH_2CH_3-PF_6^-$	Not run	3800	4450	Not run	>60	>60

*Could not be incorporated into fabric, because of thermal instability.

The data in Table 5 show several examples that show exceptional static charge dissipation performance, that is, fabrics that accepted the full
5 5000 Volt charge, and which had dissipation times under 1 second even at relative humidities of 25 percent or even 10 percent. These include Examples 23, 25, 26, 30, 32, 36, 42, and 43. All of these examples were made using antistat compounds that had thermal
10 decomposition temperatures of 370°C or higher. Comparative Example C5 showed exceptional static charge dissipation performance, but has a thermal decomposition temperature of 289°C. Comparative Example C6 with a thermal decomposition temperature of 177°C was
15 insufficiently stable for the melt-blown extrusion process. Examples 23 and 24 as well as 31 and 32 show the effect of antistat compound concentration upon static charge dissipation performance, with higher concentrations (Examples 23 and 32) showing superior
20 performance over the lower concentrations (Examples 24 and 31). Many of the examples in Table 5 that did not show good static charge dissipation performance at the 25 percent and 10 percent relative humidity levels, did show static dissipation times of less than 5 seconds at
25 50 percent relative humidity (Examples 38-41, and 44). Examples in Table 5 that did not show good static charge dissipation performance at any relative humidity at the 1% concentration may demonstrate such performance at higher levels, and/or in other polymers,
30 and/or upon annealing.

Example 47 and Comparative Example C8

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The compound of Example 1 was incorporated into polyethylene terephthalate 65-1000 melt blown fibers, which were processed into a nonwoven fabric according to the melt-blown extrusion procedure described in U.S. Pat. No. 5,300,357, column 10, which is herein incorporated by reference. For comparison, polyethylene terephthalate 65-1000 melt blown fibers without the compound were processed into a nonwoven fabric as well. The extruder used was a Brabender 42 mm conical twin screw extruder, with maximum extrusion temperature of 280°C and distance to the collector of 12 inches (30 cm).

The compound and polyethylene terephthalate 65-1000 were mixed by blending in a paperboard container using a mixer head affixed to a hand drill for about one minute until a visually homogeneous mixture is obtained. The compound was dispersed in the molten polyethylene terephthalate by mixing in the melt extrusion apparatus just prior to melt blowing. The weight percent of the compound in the urethane was 2%.

The process condition for each mixture was the same, including the melt blowing die construction used to blow the microfiber web, the basis weight of the web ($50 \pm 5 \text{ g/m}^2$) and the diameter of the microfibers (5-18 micrometers). The extrusion temperature was 280°C, the primary air temperature was 270°C, the pressure was 124 kPa (18 psi), with a 0.076 cm air gap

width, and the polymer throughput rate was about 180 g/hr/cm.

The resulting melt blown polyethylene terephthalate 65-1000 fabric made with and without the
5 compound of Example 23 was evaluated for antistatic performance using Test Method III - Static Charge Dissipation Test. The results are shown in Table 6.

Table 6. Static Charge Dissipation in Melt Blown
10 Polyethylene Terephthalate 65-1000 Fabric With and Without Octyldimethyl-2-hydroxyethylammonium bis(trifluoromethylsulfonyl)imide.

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Table 946EE00F

Example No.	Antistat Compound	Aver. Induced Electrostatic Charge (Volts)			Aver. Static Decay Rate (sec)		
		10% RH	25% RH	50% RH	10% RH	25% RH	50% RH
C8	None	Not run	3000	3100	Not run	>60	>60
47	$\text{C}_8\text{H}_{17}\text{N}^+(\text{CH}_3)_2\text{C}_2\text{H}_4\text{OH}$ $-\text{N}(\text{SO}_2\text{CF}_3)_2$ (2%)	Not run	5000	5000	Not run	0.86	3.6

The data in Table 6 shows good static charge dissipation of Example 47 compared with the control polyester fabric (Comparative Example C8).

5

Example 48 and Comparative Example C9

The compound of Example 1 was incorporated into MORTHANE™ PS440-200 urethane melt blown fibers, which were processed into a nonwoven fabric as described in Example 47, except that the extrusion temperature was 230°C. For comparison, MORTHANE™ PS440-200 urethane melt blown fibers without the compound were processed into a nonwoven fabric as well. The fabrics were tested for antistatic performance using Test Method III - Static Charge Dissipation Test. The results are shown in Table 7.

Table 7. Static Charge Dissipation in Melt Blown MORTHANE™ PS440-200 Urethane Fabric With and Without Octyldimethyl-2-hydroxyethylammonium bis(trifluoromethylsulfonyl)imide.

TABLE "BT62E.001"

Exempl e No.	Antistat Compound	Aver. Induced Electrostatic Charge (Volts)			Aver. Static Decay Rate (sec)		
		10% RH	25% RH	50% RH	10% RH	25% RH	50% RH
C9	None	Not run	5000+	5000+	Not run	>10	>10
48	C ₈ H ₁₇ N ⁺ (CH ₃) ₂ C ₂ H ₄ OH -N(SO ₂ CF ₃) ₂ (2%)	Not run	5000	5000	Not run	0.03	0.06

The data in Table 7 shows good static charge dissipation of Example 48 compared with the control polyurethane fabric (Comparative Example C9).

5

Example 49 and Comparative Example C10

The compound of Example 1 was incorporated into ASPUN™ 6806 polyethylene melt blown fibers, which were processed into a nonwoven fabric as described in
10 Example 47, except that 1 weight percent of the compound of Example 1 was used, and the extrusion temperature was 240°C. For comparison, ASPUN™ 6806 polyethylene melt blown fibers without the compound were processed into a nonwoven fabric as well. The
15 fabrics were tested for antistatic performance using Test Method III - Static Charge Dissipation Test. The results are shown in Table 8.

Table 8. Static Charge Dissipation in ASPUN™ 6806
20 Polyethylene Melt Blown Fabric With and Without Octyldimethyl-2-hydroxyethylammonium bis(trifluoromethylsulfonyl)imide.

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TABLE "ST62E00F"

Example No.	Antistat Compound	Aver. Induced Electrostatic Charge (Volts)			Aver. Static Decay Rate (sec)		
		10% RH	25% RH	50% RH	10% RH	25% RH	50% RH
C10	None	Not run	2800	3400	Not run	>60	>60
49	$\text{C}_8\text{H}_{17}\text{N}^+(\text{CH}_3)_2\text{C}_2\text{H}_4\text{OH}$ $-\text{N}(\text{SO}_2\text{CF}_3)_2$ (1%)	5000	5000	5000	0.26	0.02	0.02

The data in Table 8 shows good static charge dissipation of Example 49 compared with the control polyethylene fabric (Comparative Example C10).

5

Example 50 and Comparative Example C11

10 This example illustrates the use of an ionic liquid antistatic compound in injection molded polycarbonate. Mobay Makrolon™ LQ-3147 polycarbonate pellets were melt-injection molded using a Morgan-Press™ vertical clamp ram injection-molding machine made by Morgan industries Inc. (Long Beach, CA). The molding machine was operated at barrel and nozzle temperatures of 288°C. Molten polycarbonate (with and
15 without ionic liquid antistatic compound) was injected under pressure into a preheated aluminum mold designed to produce a flat 7.62 cm by 7.62 cm square molded part with a thickness of 0.254 cm. Two series of polycarbonate parts were produced, each series
20 comprising three parts made under identical conditions. The first series of parts were made using virgin polycarbonate resin with no additives. The second series was made by premixing the polycarbonate pellets with 1.0 weight % 1,3-ethylmethylimidazolium
25 nonafluorobutanesulfonate (Compound 12) ionic liquid antistat prior to feeding the pellets to the barrel of the ram injection-molding machine. All of the molded parts from each series were subjected to surface resistivity measurements on front and back surfaces at
30 22°C, 32% relative humidity using an ETS wide range resistance meter in Test Method IV - Surface

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Resistivity Test. The mean values of the surface resistivities for each series of parts are summarized in Table 9.

5 Table 9. Surface Resistivities of Molded Polycarbonate Sheets Without and With 1,3-Ethylmethylimidazolium Nonafluorobutanesulfonate Ionic Liquid Antistat at 1 Percent by Weight.

Example	Surface (Front or Back)	Surface Resistivity (ohms/squar e)
C11	FRONT	>10 E 12
	BACK	>10 E 12
50	FRONT	2.1 x 10 E 8
	BACK	6.7 x 10 E 7

10 It is apparent from the data in Table 9 that the addition of 1.0 weight % ionic liquid antistatic compound to the molded polycarbonate resin resulted in a dramatic decrease in the surface resistivity of the final molded part. Such reductions in surface
15 resistivity generally correlate with improved antistatic performance. Furthermore, it was noted that the addition of ionic liquid antistat to the polycarbonate had no noticeable impact on the processability of the molten polycarbonate resin or the
20 quality of the final molded articles.

Example 51 and Comparative Example 12

A polypropylene film containing antistatic compound, octyldimethyl-2-hydroxyethylammonium bis(trifluoromethanesulfonyl)imide (Example 1), was
5 prepared and evaluated for antistatic performance. For comparison, a polypropylene film without the compound was identically prepared and evaluated. Thus, the melt-blown nonwoven fabrics of Example 23 and Comparative Example C4 were pressed into films as
10 follows. About 3.4 g of the folded melt-blown fabric was placed on a steel plate within the perimeter of an 11.2 cm by 17.1 cm by 0.177 mm thick shim and covered with another steel plate. This assembly was then placed on a platen press heated to 200°C with the
15 platens nearly touching for about 30 seconds to premelt the fabric and allow for escape of air before pressing. Next, the construction was placed under 0.91 metric ton of pressure for about one minute. The assembly was removed from the press and allowed to cool for about 30
20 seconds between two unheated platens. The formed film was then removed from the shim and steel plates.

The films prepared in this way were subjected to Test Method III - Static Charge Dissipation Test. Results are shown in Table 10.

25

Table 10. Static Charge Dissipation in Escorene TM PP3505 Polypropylene Film With and Without Octyldimethyl-2-hydroxyethylammonium bis(trifluoromethylsulfonyl)imide.

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Examp e No.	Antistat Compound	Aver. Induced Electrostatic Charge (Volts)			Aver. Static Decay Rate (sec)		
		10% RH	25% RH	50% RH	10% RH	25% RH	50% RH
C12	None	Not run	Not run	4000	Not run	Not run	>10
51	C ₈ H ₁₇ N ⁺ (CH ₃) ₂ C ₂ H ₄ OH -N(SO ₂ CF ₃) ₂ (1%)	1750	1750	1400	0.003	0.007	0

The data in Table 10 shows good static charge dissipation of Example 51 compared with the control polypropylene film (Comparative Example C12).

5

Examples 52 and Comparative Example C13

Polyester film with and without a topical treatment of antistat compound were prepared and evaluated for surface resistivity. A 5 weight percent solids solution of octyldimethyl-2-hydroxyethylammonium bis(trifluoromethylsulfonyl)imide, $C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^- N(SO_2CF_3)_2$ (Example 1), in isopropanol was prepared. About 2 ml of solution was pipetted at the top of a 25.5 cm by 15.5 cm by 0.177 mm thick Mellinex 617 film. The solution was then drawn over the film using a No. 12 wire wound bar. The resulting coating was dried in a forced air oven for 2.5 minutes at 65°C. The surface resistivity of this coated film as well as an uncoated film was determined according to Test Method IV-Surface Resistivity Test. The results are shown in Table 11.

Example 53

Mellinex 617 film was coated with antistat compound, octyldimethyl-2-hydroxyethylammonium bis(trifluoromethylsulfonyl)imide, $C_8H_{17}N^+(CH_3)_2CH_2CH_2OH^- N(SO_2CF_3)_2$ (Example 1), without solvent as in Example 52, except that no solvent was used, a No. 3 wire wound bar was used to coat the compound, and the resultant coating was not dried in an oven. The surface

resistivity of this coated film was determined as in Example 52, and the results are shown in Table 11.

Various modifications and alterations of this invention will become apparent to those skilled in the art without departing from the scope and spirit of this invention.

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